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972264	28 May 1997 (28.05.97)	FI	
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(54) Title: BETAINE PRODUCT, METHOD FOR ITS MANUFACTURE, AND ITS USE

(57) Abstract

The invention relates to a solid betaine product having good treatment properties. The invention also relates to a method for the manufacture of the betaine product and its use, as well as a feed containing said betaine product and other feed components. Said solid betaine product contains a fluidity improver and/or anti-agglomeration agent, and a hydrophobic, moisture-proof layer is arranged on the surface of the particles.

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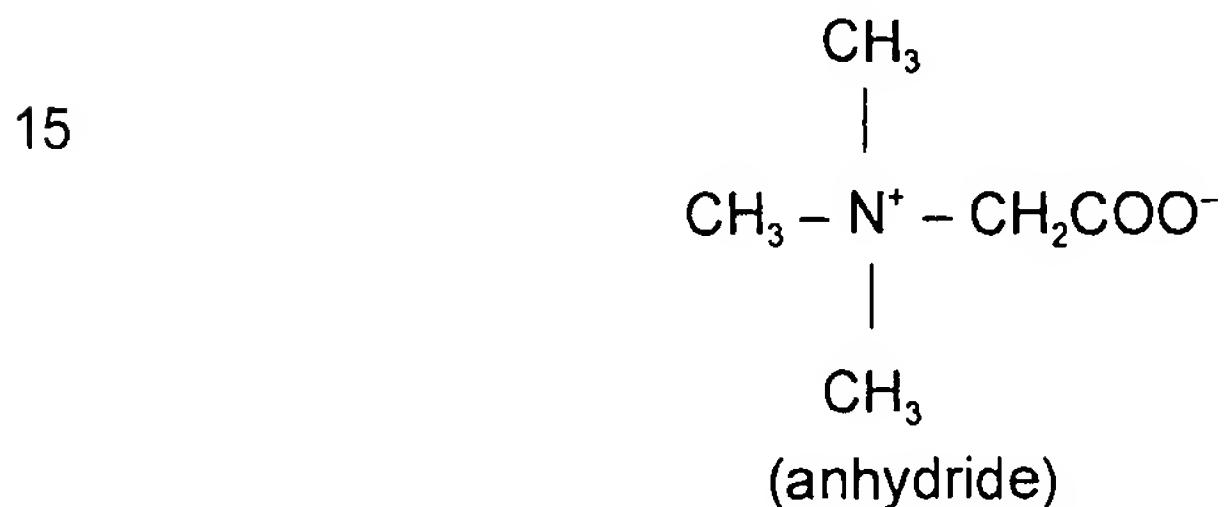
BETAINE PRODUCT, METHOD FOR ITS MANUFACTURE, AND ITS USE

FIELD OF THE INVENTION

The invention relates to a solid betaine product having good treatment properties. The invention also relates to a method for the manufacture of 5 the betaine product, and its use, as well as an animal feed containing said betaine product and other feed components.

BACKGROUND OF THE INVENTION

Betaines are fully N-methylated amino acids. Betaines are natural products that have an important function in both plant and animal metabolism. 10 One of the most common betaines is a glycine derivative in which three methyl groups are bonded to the nitrogen atom of the glycine molecule. This betaine compound is usually called betaine, glycinebetaine or trimethylglycine, and it has the following structural formula:



20

Other betaines include, for example, alaninebetaine and prolinebetaine, which has been reported, among other things, to prevent osteomalacia in chickens. A detailed description of betaines is given by R.G. Wyn Jones and R. Storey in *The Physiology and Drought Resistance in Plants*, ed. L.G. 25 Paleg and D. Aspinall, Academic Press, Sydney, Australia, 1981.

Betaine thus has a bipolar structure and it contains several chemically reactive methyl groups, which it can donate in enzyme-catalysed reactions. Most organisms are able to synthesize small quantities of betaine e.g. for the methyl function, but they are not able to react to stress by substantially 30 increasing the production and storage of betaine.

Betaine is a highly hygroscopic substance, and therefore its treatment and use as such or as part of an end product causes problems in humid conditions, since it turns viscous, lumpy and poorly flowable. In order to treat and use betaine without problems, it is necessary to protect it against humidity. 35 Hydrophilic fluidity improvers / anti-agglomeration agents (which absorb

free water) have been used previously. However, hydrophilic substances of this kind are not able to protect betaine in humid conditions, even though they improve the fluidity of betaine in good, dry conditions.

Betaine is used, among other things, as a feed additive and as a 5 crop improver of plants under stress conditions, as well as in cosmetic, pharmaceutical and food industries.

Betaine can be obtained, for example, from sugar beet by chromatographic methods. Betaine is commercially available e.g. under the registered trademark BETAFIN®, Cultor Oy, Finnsugar Bioproducts. BETAFIN® is solid 10 betaine (betaineanhydride or betainemonohydrate) produced by Finnsugar Bioproducts.

EP 0 573 876 discloses a method for the manufacture of a product containing cholinechloride by mixing cholinechloride solution in fine drops with a pulverized, hydrophobic substance, and by drying the mixture with a fluidized bed drier. Silica made hydrophobic by silanization or a metallic salt of a fatty acid can be used as the hydrophobic substance. 15

BRIEF DESCRIPTION OF THE INVENTION

Now it has been found that by means of hydrophobic substances a moisture-proof layer can be arranged on the surface of betaine particles. 20 Hence fluidity and anti-agglomeration of a betaine product, its use as such or as a part of the end-product, can substantially be improved also in humid conditions. Hydrophobic substance is preferably added to a betaine product by utilizing an efficient blender.

DETAILED DESCRIPTION OF THE INVENTION

25 The invention relates to a solid betaine product which contains a fluidity improver and/or anti-agglomeration agent. In accordance with the invention, a hydrophobic, moisture-proof layer is arranged on the surface of particles.

The solid betaine product refers to a crystalline or ground product. 30 The particle size of the crystalline product is 0.05 to 2.0 mm, preferably on the average about 0.3 to 0.6 mm. Preferably, the crystalline product does not contain practically any dustlike substance, i.e. particles with the diameter of less than about 0.01 mm. The particle size of the ground product is 1 to 100 µm.

The hydrophobic layer on the surface of the particles may consist of a metallic salt of a fatty acid, such as calcium or magnesium stearate, or hydrophobic silica. Additionally, a substance which enhances said hydrophobic substance to adhere to or spread onto the surface of betaine particles may be 5 present on the surface of the crystals.

Suitable substances that enhance the adhesion or spreading of the hydrophobic substance comprise fats, oils, fat-like substances, such as lecithins and waxes, and water-insoluble substances, such as cellulose derivatives and silicone derivatives.

10 In accordance with the invention, a new betaine product can be produced, for example, by mixing the blend of betaine and a hydrophobic, moisture-proof substance.

In the method of the invention, various mixers can be used. When only powdery hydrophobic substance is used, suitable mixers comprise various batch mixers or continuous mixers, such as drum mixers or helical/screw mixers. Mixing time depends on the power of the mixer, and also on the batch size. The object is that the hydrophobic substance mixes as evenly as possible with the betaine. In the ideal case, the powder forms a monomolecular layer on the surface of the product. Preferably, the smallest betaine crystals 20 are screened out before a treatment with the hydrophobic substance.

When a binding agent is added to the powdery hydrophobic substance, spray granulators or fluidized bed drying/coating devices can be used in the method of the invention, in addition to various batch mixers and continuous mixers.

25 Various grinders, such as an air jet grinder or a turbo mill or some other mill useful for grinding sugar crystals, can be used in the method of the invention. The betaine is thus ground with the powdery hydrophobic substance, and the obtained powdery product flows freely and it will not agglomerate.

30 In accordance with one preferred method of the invention, melted, hot (e.g. about 40 °C higher than the melting point) fat is first sprayed onto the surface of the betaine crystals. It is preferable to use a hydrogenated fat with a high melting point, whereby a product is obtained, which retains good fluidity (does not become viscous) even at tropical temperatures. Other fats/oils can 35 also be used (depending on the use of the product). When using oils that are fluid at room temperature, no heating is needed. When using fats with a high

melting point, it is preferable to preheat the betaine crystals close to the melting point of the fat. This facilitates the forming of a layer as even as possible. Thereafter the surface of the warm crystal coated with fat is 'powdered' with a hydrophobic substance. Managing the processing conditions is critical 5 (temperatures) in order that even and comprehensive protection is achieved, and the product does not stick to the walls of the equipment or elsewhere, and the product remains single, flowable crystals (no agglomeration).

Instead of spraying the fat, another technique may also be used, in which the betaine particles and the solid fat flakes, et cetera, are first mixed 10 together. Thereafter, the blend is heated to exceed the melting point of the fat while it is continuously being mixed.

The fat treatment may be carried out by using a mixer with the possibility of adding liquid (spray nozzle systems) or a spray granulator or devices of the type of fluidized bed drier/coater.

15 Useful hydrophobic substances include, for example, the following:

Metallic salts of long-chain fatty acids:

Stearates:

metallic salts of stearic acid in powder form,
(melting point in excess of 150 °C)

20 e.g. Ca-stearate, Ceasit Leviss, manufactured by Bärlocher, Germany

Ca-stearate CPR-2, manufactured by Akcros Chemicals V.O.F.,
Holland

Silica-based hydrophobic substances:

25 e.g. Sipernat D17, manufactured by Degussa Ltd., Germany

Aerosil R972, manufactured by Degussa Ltd., Germany

(and other hydrophobic Sipernat and Aerosil products)

Cellulose derivatives:

e.g. Ethocel, manufactured by Dow USA (in powder form)

30 Useful substances that enhance the adhesion or spreading of the hydrophobic substance include, for example, the following:

Fats:

35 Revel S (acetylated monoglyceride, hydrogenated soy oil); melting point 63 °C; manufactured by Loders Croklaan, Holland
Revel C; melting point 58 °C,

Revel F; melting point 46 °C,
Revel AC; melting point 37 °C,
Dynasan P 60; manufactured by Huls AG, Germany

Oils:

5 Fish oils

Lecithins:

e.g. lecithins of Metarin product line; manufactured by Lucas Mayer, Germany

Silicone derivatives (spraying in solution):

10 e.g. Pharsil MK silicone emulsion; manufactured by: Wacker Chemie GmbH, Germany

Cellulose derivatives (spraying in solution):

e.g. Ethocel; manufactured by Dow USA

15 **Example 1**

Betaine anhydride (Nutristim, manufactured by Cultor Oy; quantity 50 kg, granule size 0.27 to 1.0 mm) was introduced into a drum mixer (Forberg F-60, manufactured by Fa. Halvor Forberg A/S, Norway) and 3 kg calcium stearate (Ceasit Leviss, manufactured by Bärlocher, Germany, particle size 20 99% < 71 µm) was added thereto. Mixing was carried out at room temperature for about three minutes. The obtained product had good fluidity, and it remained fluid for at least three hours at a temperature of 30 °C, the relative air humidity being 95%.

Example 2

25 Into a continuous screw mixer (length of the mixing worm 4 m, diameter 400 mm, two mixing points in the screw, manufactured by Siirtooruvi Oy, Finland) were fed betaine anhydride (Nutristim, manufactured by Cultor Oy; granule size 0.27 to 1.0 mm) 5,000 kg/hour and 6% calcium stearate (Ceasite Leviss, manufactured by Bärlocher, Germany; particle size 30 99% < 71 µm) through an additive dispenser (Accurate, manufactured by Accurate Dry Material Feeders Inc., USA). Mixing was carried out continuously in the mixing helix/screw. Temperature was 20 °C and duration of mixing in the mixing helix was two minutes. The obtained product had good fluidity and it remained fluid at least for three hours at a temperature of 30 °C, the relative air humidity being 35 95%.

Example 3

Betaineanhydride (Nutristim, manufactured by Cultor Oy; quantity 47.5 kg, granule size 0.27 to 1.0 mm) was introduced into a drum mixer (Forberg F-60, manufactured by Fa. Halvor Forberg A/S, Norway) and preheated to 55 °C. Thereafter 2.5 kg hot (100 °C) fat (Revel-S, manufactured by Loders Croklaan, Holland) was added by spraying (nozzle size 4004, pressure 3 bar) and simultaneously mixing (for 1.3 minutes). Finally, 1 kg calcium stearate (Ceasit Leviss, manufactured by Bärlocher, Germany; particle size 99% < 71 µm) was added, and the blend was cooled to 30 °C and mixed for 3.7 minutes. The obtained product's solubility in water was poor and fluidity was good: it remained fluid at least for three hours at a temperature of 30 °C, the relative air humidity being 95%.

Example 4

The test was conducted in the same way as in Example 1, but instead of calcium stearate, hydrophobic silica (Sipernat D 17, manufactured by Degussa, Germany; average granule size 10 µm) was used. The obtained product was very fluid, and it remained fluid at least for three hours at a temperature of 30 °C, the relative air humidity being 95%.

Example 5

The test was conducted in the same way as in Example 3, but instead of calcium stearate, hydrophobic silica (Sipernat D 17, manufactured by Degussa, Germany; average granule size 10 µm). The obtained product's solubility in water was poor and fluidity was good: it remained fluid at least for three hours at a temperature of 30 °C, the relative air humidity being 95%.

Example 6

Betainemonohydrate (technical quality, manufactured by Cultor Oy; quantity 47.5 kg, granule size 0.25 to 1.25 mm) was introduced into a drum mixer (Forberg F-60, manufactured by Fa. Halvor Forberg A/S, Norway) and preheated to 55 °C. Thereafter, 1.5 kg hot (100 °C) fat (Revel-S, manufactured by Loders Croklaan, Holland) was added by spraying (nozzle size 4004, pressure 3 bar) and simultaneously mixing (for 1.3 minutes). Finally, 1.5 kg calcium stearate (Ceasit Leviss, manufactured by Bärlocher, Germany; particle size 99% < 71 µm) was added, and the blend was cooled to 30 °C and mixed for 3.7 minutes. The obtained product's solubility in water was poor and fluidity was good.

Example 7

The test was conducted in the same way as in Example 6, but instead of calcium stearate, hydrophobic silica (Sipernat D 17, manufactured by Degussa, Germany; average particle size 10 µm) was used. The obtained product had good fluidity and it remained fluid at least for three hours at a temperature of 30 °C, the relative air humidity being 95%.

Example 8

Betaineanhydride (Betafin BP, manufactured by Cultor Oy; particle size 0.05 to 1.0 mm) was fed into a continuous air jet grinder (Pulva FP Micro-nization, manufactured by Oy Finnpulva Ab); simultaneously, hydrophobic silica (Aerosil R 972, manufactured by Degussa; average particle size 16 nm) was also fed into the grinder. The total quantity of betaineanhydride was 400 kg and that of silica 4 kg (1%). The average grinding fineness of the obtained product was less than 20 µm, the product was very fluid and it did not agglomerate.

CLAIMS

1. A solid betaine product which contains a fluidity improver and/or anti-agglomeration agent, **characterized** in that a hydrophobic, moisture-proof layer is arranged on the surface of particles.
- 5 2. A product as claimed in claim 1, **characterized** in that the layer consists of a metallic salt of a fatty acid, such as calcium or magnesium stearate.
3. A product as claimed in claim 1, **characterized** in that the layer consists of hydrophobic silica.
- 10 4. A product as claimed in claim 1, 2 or 3, **characterized** in that a substance which enhances the hydrophobic substance to adhere or to spread is additionally arranged on the surface of the crystals.
- 5 15 5. A product as claimed in claim 4, **characterized** in that the substance which enhances the hydrophobic substance to adhere or to spread is selected from the group of fat, oil, fat-like substances, such as lecithins and waxes, and water-insoluble substances, such as cellulose derivatives and silicon derivatives.
6. A method for the manufacture of a crystalline betaine product as claimed in claim 1, 2 or 3, **characterized** in that the solid betaine and 20 the hydrophobic substance are mixed together.
7. A method as claimed in claim 6, **characterized** in that the solid betaine is mixed with a metallic salt of a fatty acid, such as calcium or magnesium stearate.
8. A method as claimed in claim 6, **characterized** in that the 25 solid betaine is mixed with hydrophobic silica.
9. A method as claimed in claim 1, 2 or 3, **characterized** in that the betaine is ground with the hydrophobic substance.
10. A method for the manufacture of a crystalline betaine product as claimed in claim 4 or 5, **characterized** in that oil or melted fat is first arranged on the surface of the betaine crystals, whereafter a hydrophobic substance is arranged on the surface.
11. A method as claimed in claim 10, **characterized** in that the melted fat is a hydrogenated fat with a high melting point, and the betaine crystals are preheated close to the fat melting point.
- 35 12. A method for the manufacture of a betaine product as claimed in claim 4 or 5, **characterized** in that, first, the betaine crystals and the

solid fat are mixed together, whereafter the hydrophobic substance, such as a metallic salt of a fatty acid or hydrophobic silica, is added, and the blend is heated to exceed the fat melting point while it is continuously being mixed.

13. Use of a product as claimed in any one of claims 1 to 5 as an
5 animal feed additive.

14. Use of a product as claimed in any one of claims 1 to 5 in fertil-
izers.

15. Use of a product as claimed in any one of claims 1 to 5 in cos-
metic skin care products or in toiletries.

10 16. Use of a product as claimed in any one of claims 1 to 5 in a
pharmaceutical product or in a product of unverified pharmacological effects.

17. Use of a product as claimed in any one of claims 1 to 5 in a food
product.

18. An animal feed, **characterized** by containing betaine
15 product as claimed in any one of claims 1 to 5 and other feed components.

INTERNATIONAL SEARCH REPORT

International application No.

PCT/FI 98/00449

A. CLASSIFICATION OF SUBJECT MATTER

IPC6: B01J 2/30, A23K 1/02, A23P 1/06

According to International Patent Classification (IPC) or to both national classification and IPC.

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC6: A23K, A23L, A23P, B01J, A61K

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

SE, DK, FI, NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

WPI

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WPI/Derwent's abstract, No 75-43835W, week 7526, ABSTRACT OF JP, 438407 (AS KIRG ORG CHEM), 28 January 1975 (28.01.75), abstract	1,6,9,13,18
Y	--	2-3,7-8, 14-17
X	WPI/Derwent's abstract, No 78-83702A, week 7825, ABSTRACT OF JP, 586889 (AS KIRG ORG CHEM), 6 January 1978 (06.01.78), abstract	1,6,13,18
Y	--	2-3,7-8, 14-17

 Further documents are listed in the continuation of Box C. See patent family annex.

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1 Sept 1998

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C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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INTERNATIONAL SEARCH REPORT

Information on patent family members

27/07/98

International application No.

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